Titration Lab

Titration involves measuring the exact volume of a solution of known concentration that is required to react with a measured volume of a solution of unknown concentration, or with a weighed sample of unknown solid. A solution of accurately known concentration is called a standard solution. Solutions of sodium hydroxide are commonly used in titration analyses of samples containing an acidic solute. Although sodium hydroxide is a solid, it is not possible to prepare standard sodium hydroxide solutions by weight. Solid sodium hydroxide is usually of questionable purity. In addition, sodium hydroxide reacts with carbon dioxide from the atmosphere and is also capable of reacting with the glass of the container in which it is provided. For these reasons, sodium hydroxide solutions are generally prepared to be approximately a given concentration. They are then standardized by titration of a weighed sample of a primary standard acidic substance (primary meaning it contains only one acidic proton). In this experiment, potassium hydrogen phthalate (KHP) will be the primary standard used. By measuring the volume of the sodium hydroxide solution necessary to react completely with a weighed sample of KHP, the concentration of the sodium hydroxide can be calculated.

In titration analyses, there must be some means of knowing when enough titrant has been added to react completely with the sample being titrated. In an acid/base titration analysis, there should be an abrupt change in pH when the reaction is complete. There are various natural and synthetic dyes called indicators that exist in different colored forms at different pH values. A suitable indicator can be chosen that will change color at a pH value consistent with the point at which the titration reaction is complete. The indicator to be used in this experiment is phenolphthalein, which is colorless in acidic solutions, but changes to a pink form at basic pH. The point at which this color change has occurred is called the end point. It specifies at which point the titrant and analyte have been combined in equal molar amounts.

In this lab you will first use KHP to standardize a solution of sodium hydroxide you make. Then you will use that solution to determine the concentration of hydrochloric acid solution that has been given to you. Finally, you will perform a titration to determine the molar mass of a solid acidic solute.

Apparatus and Materials

burette	burette clamp	5-mL pipette and bulb
soap	~0.1M NaOH	potassium hydrogen phthalate (KHP)
phenolphthalein	unknown acid solution	distilled water
wash bottle	weighing boats	watch glass
ring stand	2 50-mL beakers	3 Erlenmeyer flasks (250 mL)
unknown solid acid		

Procedure

Part 1 Standardizing the NaOH Solution

1. Prepare 1000 mL of a solution of sodium hydroxide with a concentration of approximately 0.1 M. Make sure to use a volumetric flask and distilled water. Follow proper protocols for making solutions.

2. Obtain and clean a burette and a pipette with soap, water, and a brush. Rinse with tap water, and then with distilled water. Water should run down the inside of burette and pipette in sheets and should not bead up anywhere on the interior of the glassware. Finally, rinse the burette with 5-10 mL of the sodium hydroxide solution. Rinse the pipette with a small amount of the unknown acid solution just prior to use. (A burette or pipette should always be rinsed with the solution with which it will be filled just prior to filling it with the solution, in order to avoid diluting the solution)

3. Clean three 250-mL Erlenmeyer flasks with soap, water, and a brush. Rinse with tap water, and then with a small portion of distilled water. Label the flasks 1, 2, and 3.

4. Mass three samples of KHP of approximately 0.6 g. The KHP is stored in the desiccator to ensure that there is no water contaminating the sample. Record the exact mass of each KHP sample to as many digits as possible. Be certain not to confuse the samples.

5. Add approximately 100 mL (exact volume is not relevant) of water to each KHP sample, along with 2-3 drops of phenolphthalein indicator solution. Swirl to dissolve the KHP samples completely.

6. Fill the burette with sodium hydroxide solution. Record the initial reading of the NaOH solution in the burette to the nearest 0.01 mL, remembering to read across the bottom of the meniscus.

7. Begin adding NaOH solution from the burette to the sample in the Erlenmeyer flask, swirling the flask constantly during the addition. As the NaOH solution enters the solution in the Erlenmeyer flask, streaks of red or pink will be visible. They will fade as the flask is swirled. Eventually the red streaks will persist for a longer and longer period of time. This indicates the approach of the endpoint of the titration.

8. Begin adding NaOH one drop at a time, with constant swirling, until one single drop of NaOH causes a permanent pale/faint pink color that does not fade on swirling for at least 30 seconds. Record the reading of the burette to the nearest 0.01 mL.

9. Repeat the titration of the remaining two KHP samples. Record both initial and final readings of the burette to the nearest 0.01 mL. If any sample is over-titrated (the pink color is dark rather than pale), the sample must be repeated.

10. Given that the molar mass of KHP is 204.22 g/mol, and that it reacts in a 1:1 ratio with sodium hydroxide, calculate the molarity of your sodium hydroxide solution from each trial. Use as many significant figures as possible. Calculate an average molarity, which will be used in the following determinations.

11. Dispose of all neutralized solutions down the drain with copious amounts of water.

Determination of the Molarity of a Hydrochloric Acid Solution

1. Clean and dry a small beaker, and obtain 25-30 mL of the hydrochloric acid solution. Cover the solution with a watch glass to avoid evaporation.

2. Clean three Erlenmeyer flasks, and label as samples 1, 2, and 3. Rinse the flasks with small portions of distilled water.

3. Using the rubber bulb, rinse the 5-mL pipette with small portions of the hydrochloric acid solution. Pipette exactly 5 mL of the solution into each of the Erlenmeyer flasks. Add approximately 100 mL of distilled water to each flask, as well as 2-3 drops of phenolphthalein solution.

4. Titrate each sample with NaOH from the burette as before.

5. Based on the volume of the unknown acid sample taken, and on the volume and average concentration of the NaOH solution used, calculate the concentration of the hydrochloric acid solution.

6. Dispose of all neutralized solutions down the drain with copious amounts of water.

Determination of the Molar Mass of an Unknown Solid Acid

1. Determine the molar mass of the unknown solid. You will write your own procedure and determine your own materials for this experiment.

Data and Observations

Collect and record all relevant data for the experiment. Be sure to use the proper significant figures, uncertainties, and units.

Analysis of Data

Perform all of the calculations necessary to determine the experimental molarity of your NaOH solution, experimental molarity of the unknown hydrochloric acid solution, and molar mass of the unknown acid. Ask your instructor for the theoretical molarity of the unknown acid solution.

Conclusion

Calculate your percent error for the unknown determination, and give some possible explanations for your error. Be specific! State what you learned and analyze the procedure. Identify the strengths and weaknesses of your procedure. Make any suggestions to improve the experiment.

Lab Report

The lab report should contain a brief introduction, a complete procedure for the lab, data and observations, data analysis, and full conclusion.